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# National Hydrology Research Institute
# Institut national de recherche en hydrologie

Environment CANADA Environnement
2056174K VOL 87 ISS 11 870600
EXC # 001
GB 707 N42
NHRI CONTRIBUTION
OOFF

Inland Waters and Lands Directorate
Saskatoon, Saskatchewan, Canada

Direction generale des eaux interieures et les terres
Saskatoon, Saskatchewan, Canada

Canada

ISSN 0838-1992

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# ALUMINUM CONTAMINATION OF GROUND WATER PART II: SPRING MELT IN CHALK RIVER AND TURKEY LAKES WATERSHEDS --- PRELIMINARY RESULTS

*Unpublished Report*

NHRI Contribution No. 87011

June 1987

## MANAGEMENT PERSPECTIVE

One of the effects of acid precipitation is to liberate and to mobilize aluminum from sub-surface geological materials. Elevated aluminum concentrations in surface waters have been linked to fish mortality and deformities, especially during the spring melt period. The severity of the biological effects depends on the form of the aluminum complex present in the water. Although changes in the concentrations of the different complexes of aluminum have been recorded in surface water, few measurements exist for the same complexes in ground water. Ground water plays an important role in determining surface water chemistry, so that the concentrations and forms of aluminum in ground water must be determined in order to understand the interaction of surface and ground waters in terms of aluminum.

Two watersheds with seasonally acidified ground water were examined to determine the changes in concentration of the various aluminum complexes during the spring melt period. Although both watersheds receive precipitation of similar acidifying potential (30 kg $ha^{-1}$ $y^{-1}$), there are differences in the distribution and concentration of the aluminum complexes. In both locations, it appears that the controlling factors are the pH and the carbonate content of the overburden. The influence of ground water -borne aluminum on the surface water into which it flows is still under investigation.

## PERSPECTIVE DE GESTION

Les précipitations acides ont notamment pour effet de libérer et de mobiliser l'aluminium des matériaux géologiques souterrains. Des rapports ont été établis entre les concentrations élevées d'aluminium dans les eaux de surface, surtout au cours de la fonte des neiges au printemps, et des cas de mortalité et de difformité chez les poissons. La gravité des effets biologiques tient à la forme du complexe d'aluminium dans l'eau. Bien que des fluctuations de la concentration des divers complexes d'aluminium aient été constatées dans l'eau de surface, il n'existe à peu près pas de mesures sur ces complexes dan les eaux souterraines. L'eau souterraine est très importante pour déterminer la composition chimique de l'eau de surface; pour cette raison, les concentrations d'aluminium dans l'eau souterraine et les formes sous lesquelles il se trouve doivent être déterminées afin de comprendre l'interaction entre les eaux de surface et les eaux souterraines en ce qui concerne l'aluminium.

On a étudié deux bassins hydrographiques où on trouve, selon la saison, de l'eau souterraine acidifiée afin d'établir les changements de concentration des divers complexes d'aluminium au cours de la période de fonte des neiges au printemps. Même si les deux bassins recoivent des précipitations ayant un potentiel acidifiant similaire (30 kg $ha^{-1}$ $a^{-1}$), il existe des différences sur le plan de la distribution et de la concentration des complexes d'aluminium. Dans un bassin comme dans l'autre, il semble que le pH et la teneur en carbonates de la couche de terrain sus-jacente soient les facteurs qui régissent le phénomène; on étudie toujours l'influence de l'aluminium charrié par l'eau souterraine sur l'eau de surface où elle se déverse.

Abstract

Two Watersheds with seasonal pH depressions in the groundwater exhibit high aluminum concentrations associated with these acidification events. The Watersheds receive similar atmospheric loadings of $H^+$ and $SO_4^{2-}$ but are covered by different surficial deposits. The aluminum is measured in four fractions by varying the pH and time conditions during analysis. The resulting classes calculated from these fractions are Class a, containing fast reacting monomeric aluminum, predominatly inorganic $Al^{3+}$ and hydroxy compounds; Class b, containing fast reacting complex aluminum species, mainly inorganic $F^-$ complexes and simple organic complexes; Class c, containing slow reacting amorphous and some polymeric aluminum species, and a class containing a residue of unknown aluminum species adding to the total acid extractable aluminum. There are significant differences in the distribution of the various aluminum species between the two watersheds, both with depth and in the predominant class identified. The generally higher values measured at one watershed are consistant with the lower carbonate content of the overburden. In both locations it appears that the controlling factor is the pH of the groundwater, with all but Class c species increasing with decreasing pH. The difference between the distributions of aluminum species in groundwater and surface water is under investigation to determine the influence of groundwater-bourne aluminum on the surface water into which it flows.

Résumé

Deux bassins hydrographiques dont les eaux souterraines subissent des baisses saisonnières du pH contiennent de fortes concentrations d'aluminium résultant de ces acidifications. Les bassins hydrographiques reçoivent les mêmes charges atmosphériques de $H^+$ et de $SO_4^{2-}$, mais ils sont recouverts par des formations superficielles différentes. On a mesuré l'aluminium dans quatre fractions en faisant varier le pH et le facteur temps pendant l'analyse. Les catégories établies à partir de ces fractions sont les suivantes: catégorie a, çontenant de l'aluminium monomère à réaction rapide, principalement de l'$Al^{3+}$ inorganique et des composés hydroxylés; catégorie b, contenant des types d'aluminium complexes à réaction rapide, principalement des complexes $F^-$ inorganiques et des complexes organiques simples; catégorie c, contenant des types d'aluminium amorphes à réaction lente et certains types d'aluminium polymériques; et une catégorie contenant un résidu d'un type d'aluminium inconnu qui s'ajoute à l'ensemble de l'aluminium extractible au moyen d'acides. Il existe des différences importantes au chapitre de la distribution des divers types d'aluminium entre les deux bassins hydrographiques, à la fois dans la profondeur et la catégorie prédominante identifiée. Les valeurs généralement plus élevées mesurées à l'un des bassins hydrographiques correspondent à la faible teneur en carbonates de la surcharge. Dans les deux endroits, il semble que le pH des eaux souterraines soit le facteur clé: tous les types d'aluminium, sauf ceux de la catégorie c, augmentent lorsque le pH baisse. On étudie actuellement la différence entre la distribution des types d'aluminium dans les eaux souterraines et les eaux de surface afin de déterminer l'influence de l'aluminium contenue dans les eaux souterraines sur les eaux de surface dans lesquelles il se déverse.

## Introduction

Acidic precipitation has led to acidification of streams and lakes in the Canadian Shield area of Eastern North America. Increases in aluminum concentration have been recorded in these surface waters, especially during periods of depressed pH values (Schofield and Trojnar, 1980; LaZerte, 1984; Driscoll et al., 1980; Campbell et al., 1984; and Johnson et al, 1981). The instances of groundwater acidification with coincident high aluminum levels are less frequently documented in the literature (Craig and Johnston, 1987; Hulteberg and Johansson, 1981; and Anderson and Bowser, 1986). Groundwater is important in the overall hydrologic cycle because groundwater pH and aluminum concentrations influence surface water pH and aluminum concentrations (Craig and Johnston, 1987; and Corbett, 1982).

Consequently, two watersheds with seasonal pH depressions in groundwater were investigated to determine whether high aluminum concentrations were associated with these acidification events. The two areas studied receive similar atmospheric loadings of $H^+$ and $SO_4^{2-}$, but the surficial deposits are quite different. A comparison of the two watersheds lead to a better understanding of the overall effect of acidic precipitation on groundwater pH and aluminum concentrations.

Aluminum exists in many complexes which have different mobilities and biological influences. Under the circumstances, it is not sufficient to determine only the total aluminum concentration when assessing either the mobility of aluminum ions in the environment or their effects. Although the chemistry of the surface water will ultimately determine the effect on biological systems (Grahn, 1980; Cronan and Schofield, 1979), it is important to know which forms of aluminum are present in the groundwater, and in what concentrations, not only in order to understand the mobility of aluminum in the subsurface, but also to determine which forms are contributed to the surface water system. It would be important to know , for example, whether the monomeric form or simple hydroxy ion species of aluminum were being contributed by groundwater, because these have been identified as the most toxic to fish (Driscoll et al., 1980; and Thellen and Van Collie, 1983).

To distinguish the different forms, various schemes for measuring aluminum complexes have been proposed ( LaZerte, 1984; Hendershot et al., 1984 and James et al., 1983). The method proposed by Hendershot et al. (1984) was adapted by Chew (1986) for use in the ground water environment. Hendershot et al. used their method to study the soil aluminum concentrations at three sites in the Laurentians, but their investigations were restricted to the soil zone above the water table. The present work attempts to extend the measurements of aluminum speciation into the water table.

Thus, the purpose of the study discussed in this paper is twofold: to measure the aluminum concentrations in groundwater in areas with seasonally depressed pH values, and to quantify the different forms of aluminum present in these groundwaters and associated surface waters in order to understand both its mobility and its potential effect on aquatic life.

## Site Description

The two watersheds studied are located in regions with very different surficial geology. The Turkey Lakes Watershed, near Sault Ste. Marie, Ontario, is located on the Canadian Shield in an area with relatively shallow overburden. The Perch Lake Basin, near Chalk River, Ontario, is located in the thick sands and gravels of the Upper Ottawa River Valley and no bedrock is exposed in the immediate area. The two regions receive rain of similar pH (average 4.2) and similar $SO_4^{2-}$ loadings (~30 kg $ha^{-1}$ $y^{-1}$), but differ markedly in the carbonate content of the overburden.

The Turkey Lakes Watershed is situated about 50 km north of Sault Ste. Marie (Figure 1). The bedrock is composed of greenstones and granites consisting mainly of hornblende, plagioclase, chlorite, and epidote, with minor amounts of quartz, calcite, pyrite and magnetite (Craig and Johnston, 1983; Armbrust, 1969). Within the watershed, the bedrock is overlain by till of varying thickness (0 to 70 m). The carbonate content of the tills varies from 0 to 5 %. At the two detailed study sites, the till is predominantly fine grained silty clay with many large boulders. The carbonate content averages 0.2% and increases with depth. All the sampling points are located in the till units overlying the bedrock.

The Chalk River study site is situated 200 km northwest of Ottawa (Figure 2). The thick overburden consists mostly of fine grained and well sorted sand units overlying compacted till of varying thickness. The sand units contain a small percentage of silt and clay and are composed of quartz or feldspar with smaller amounts of mica, chlorite, and hornblende. The concentrations of sulfide, organic matter, vermiculite, and iron and manganese oxyhydroxides in total are less than 1%. Trace quantities of calcite are visible under the optical microscope. A more detailed description of the overburden is given in Jackson and Inch (1980). All the sampling points are located in the permanent water table within the uppermost sand unit.

## Installations

In the Turkey Lakes Watershed, the two sites for the aluminum speciation study consist of groups of single piezometers installed at depths less than 4 metres (HU - H1, and W3 in Figure 1). Wells at HU - H1 are located on the same hillside and are identified as HU 0.67, H1-1.81, H1-2.78, and H1-3.70, where the suffix represents the depth of the well screen. Surface water was sampled at the weir (CFS 47) on the stream closest to this site and the results used for purposes of comparison. At W3 the wells are W3-1.2, W3-2.2, and W3-3.74, where the suffix again denotes the depth of the well screen. Detailed information regarding the test wells is given in two publications (Craig and Johnston, 1983; and Craig and Johnston,1987).

In the Chalk River Area, multi-level peizometers were installed (Pickens et al., 1978). The depths for the six piezometers at HA (HA2 through HA7) were 2.5 to 5.0 metres at 0.5 metre intervals. At LJ6, the six piezometers, LJ6-2 through LJ6-7, are spaced at one-metre intervals between 2 and 7 metres below the surface.

## Methods and Materials

### Field

The groundwater samples were obtained from the piezometers using a portable peristaltic pump. The pH, temperature, and specific conductance were measured in the field or, within one half hour, in a field laboratory. Samples appeared to be clear and colourless, and were analyzed without filtration. All samples for aluminum speciation were analyzed in the field or in a nearby field laboratory, usually within 20 minutes. Samples for total acid extractable aluminum were collected, acidified in the field, and then shipped to Ottawa where they were analysed within four or five days of collection.

During 1985 in the Turkey Lakes Watershed, 25 samples were collected at HU -H1 site: 5 in April, 8 in June, 5 in August, and 7 in November. During June and August, 8 samples were collected at W3. For HU 0.67 which is very shallow and above the permanent water table, samples could only be obtained during periods of temporary saturation such as following spring melt or following prolonged periods of rain. The remaining wells are in the permanent water table, although the sample volume recovered was often quite small.

At Chalk River, 51 samples were collected during the spring melt period. Eleven were collected in March, 15 in April just before melt, 12 at the peak of the melt, and 12 after the peak event. These wells are all located in the permanent water table and produce water year around.

Analyses

The methodology for separating the aluminum into four different groups of species is discussed in an earlier report (Chew, 1986). The analytical technique is based on controlling the reaction with oxine under specific pH and time conditions. By varying these two conditions, three fractions plus a total aluminum concentration are determined. From these three fractions and the total aluminum, three classes of aluminum are calculated, based on their reactivity in the chemical environment.

The operating conditions for obtaining the three fractions and a total value are as follows: fraction 1 - pH 5.0, extraction for 15 seconds; fraction 2 - pH 8.3, extraction for 15 seconds; fraction 3 - pH 8.3, extraction for 60 seconds; and total acid extractable - same conditions as fraction 3 following acidification with 1% nitric acid for four days.

From the results of these analyses, the three classes of species are calculated as follows: Class a, equivalent to fraction 1, contains fast reacting monomeric aluminum, predominantly inorganic $Al^{3+}$ and hydroxy compounds; Class b, the difference between fraction 2 and fraction 1, contains fast reacting complex aluminum species, mainly inorganic F complexes and simple organic complexes; and Class c, the difference between fraction 3 and fraction 2, contains slow reacting amorphous and some polymeric aluminum species. The total acid extractable aluminum is reported as total aluminum, and the difference between the total and fraction 3 contains unknown aluminum species.

During the course of the experiment, three spectrophotometers were used, a Spectronics 340 (Bausch and Lomb), a battery operated Spectronic Mini 20, and an LKB Ultospec II model 4050. Care was taken to ensure the compatibility of the results obtained using the three instruments. Based on multiple analyses of samples, results from the three instruments are reproducible to $\pm 10 \mu g\ L^{-1}$.

The results using the oxine method were compared with those obtained using Atomic Absorption Spectrophotometry (AAS). For standard samples, prepared using aluminum chloride, aluminum oxalate, and aluminum citrate, the values were identical. For six samples in the 100-500 $\mu g\ L^{-1}$ Al range from the Turkey Lakes Watershed, the values using the oxine method were 10% higher than those using AAS. Comparison of the two methods (Hendershot, Department of Renewable Resources, Macdonald College of McGill University, Ste. Anne de Bellevue, Quebec, H9X 1C0 pers. comm. 1987) suggests that the oxine method over estimates the total aluminum values by 10 to 20%. The comparison of the relative concentration, however, remains valid for the two sites.

## Results and Discussion

At both the Turkey Lakes study sites, the concentrations of the total aluminum and of all the classes vary with depth. At HU - H1 (Figure 3), the highest total aluminum concentrations (700µg $L^{-1}$) occur in the shallow groundwater, less than 1 metre in depth, and the concentrations then decrease rapidly until only trace amounts were measured below 3 metres. The total Al concentration in the adjacent surface water stream (400 µg $L^{-1}$) is intermediate between that of the shallow and that of the deeper groundwater. The unidentified aluminum species (total aluminum - Fraction 3) dominate the groundwater samples, while in the surface water samples the Class a ($Al^{3+}$ and hydroxy compounds) dominate. The Class b species are approximately the same in the shallow groundwater and the surface water. The different distributions of the various classes in groundwater and surface water could be significant, as Class a species are considered to be the most toxic to aquatic life. Similar results were obtained at the second site, W3.

At Chalk River, variations in aluminum concentrations with depth were also recorded at both sampling sites. At HA (Figure 3), the total aluminum concentrations increase from 165µg $L^{-1}$ at 2.5 metres to over 1580µg $L^{-1}$ at 4.0 metres, before decreasing to 300µg $L^{-1}$ at 5.0 metres. Most of this variation is due to changes in the concentration of Class a species, and to a lesser degree in the concentration of Class b species (inorganic $F^{-}$ and organic complexes). Class c concentrations remain low and relatively constant.

At LJ6, a different pattern is evident in the variation of total aluminum concentration with depth. The highest concentrations occur at 2.0 metres and between 5.0 and 7.0 metres, and the lowest value is at 3.0 metres. Again, most of the variation is attributed to changes in Class a and Class b species. Although both sampling sites are in a groundwater recharge zone, different flow regimes operate at each site, and the differences in flow contribute to the distribution of values observed. The highest total Al values at these two sites are 1600 and 650µg $L^{-1}$, respectively.

A comparison of values from the two watersheds shows that the major difference lies in the location of the highest aluminum concentrations in the groundwater profile. In the Turkey Lakes the maximum values occur near the surface at both sampling sites; at Chalk River the highest values occur at greater depths even when a high value occurs near the surface, as at LJ6. Furthermore, the total aluminum concentrations recorded at the Turkey Lakes Watershed are generally lower than those recorded at Chalk River. There are also major differences in the relative proportions of the various species, with the unidentified species (total - Fraction 3) dominating the Turkey Lakes Watershed samples, and Classes a and b dominating at Chalk River.

A comparison of pH and the concentrations of various aluminum species in the Turkey Lakes Watershed (Figure 4) shows a variety of trends. The total aluminum increases rapidly with decreasing pH in the range 5.8 to 4.9, as would be expected. The major change in concentration occurs in the unidentified, complex organic and refractory aluminum group, the difference between total acid extractable and the fraction 3 aluminum species. There are much smaller increases in Class a ($Al^{3+}$ and hydroxy compounds) and Class b (inorganic $F^-$ and organic complexes) species over the same pH range. The Class c (slow reacting amorphous and some polymeric aluminum species) concentration remains approximately constant.

The variation in aluminum concentration with ground water pH was also examined at the two Chalk River sites. At HA (Figure 4) the concentrations of total acid extractable, Class a, and Class b species all increase with decreasing pH in the range under study (5.8 to 5.2), with a major increase occurring at pH values between 5.3 and 5.2. The concentration of Class c species remains relatively constant at all pH values. At the second Chalk River site (LJ6) the concentrations of all classes of aluminum increase slightly with decreasing pH. In contrast to the previous site, when the pH range (5.3 to 5.2) is reached, the concentrations increase less dramatically (~25%).

At both watersheds, the groundwater concentrations of total aluminum and of Class a and Class b species all increase with decreasing pH. At Chalk River, the concentrations of the total and Class a species increase markedly in the pH range 5.3 to 5.2, but this is not the case at the Turkey Lakes sites. The concentrations of the Class c species remain relatively constant with varying pH at both watersheds.

While a preliminary interpretation of the results from the two watersheds is possible with respect to depth and pH, the sample collections were too widely spaced in time to permit more than a preliminary discussion of the temporal variations in aluminum concentration. At Chalk River, the highest recorded values of total aluminum occur in mid-April, at a depth of 3.5 metres (Figure 5). This maximum corresponds to the start of spring melt. The highest concentration of Class b species occurred after spring melt. The concentration of Class c species is very low and varies throughout the period.

In summary then, although both watersheds receive precipitation of similar acidifying potential, indicated by comparable pH values (average 4.2) and $SO_4^{2-}$ loading ( 30 kg $ha^{-1}$ $y^{-1}$ ), there are significant differences in the distribution of aluminum species. The differences betweem watersheds in the variation of aluminum concentration with depth, as well as the differences within the watersheds, are related both to the

different surficial deposits and to the particular groundwater flow patterns at each of the sites. In both locations, however, it appears that the controlling factor is the pH of the ground water, with all but the Class c species increasing with decreasing pH.

The generally higher values for aluminum measured at Chalk River are consistent with the lower carbonate content in the overburden and the lower pH in the groundwaters. A discussion of the mechanisms controlling aluminum mobility at the two watersheds, however, will have to await the results of more detailed sampling and analysis, particularly at the Turkey Lakes Watershed. The distribution of the different classes of aluminum species at the two sites appears to vary both in space and time, and these variations will be taken into account during future sampling programs.

The difference between the distributions of aluminum species in groundwater and surface water, which is shown by the results at HU -H1 and CFS 47, is also under investigation to determine the influence of groundwater-borne aluminum on the surface water into which it flows.

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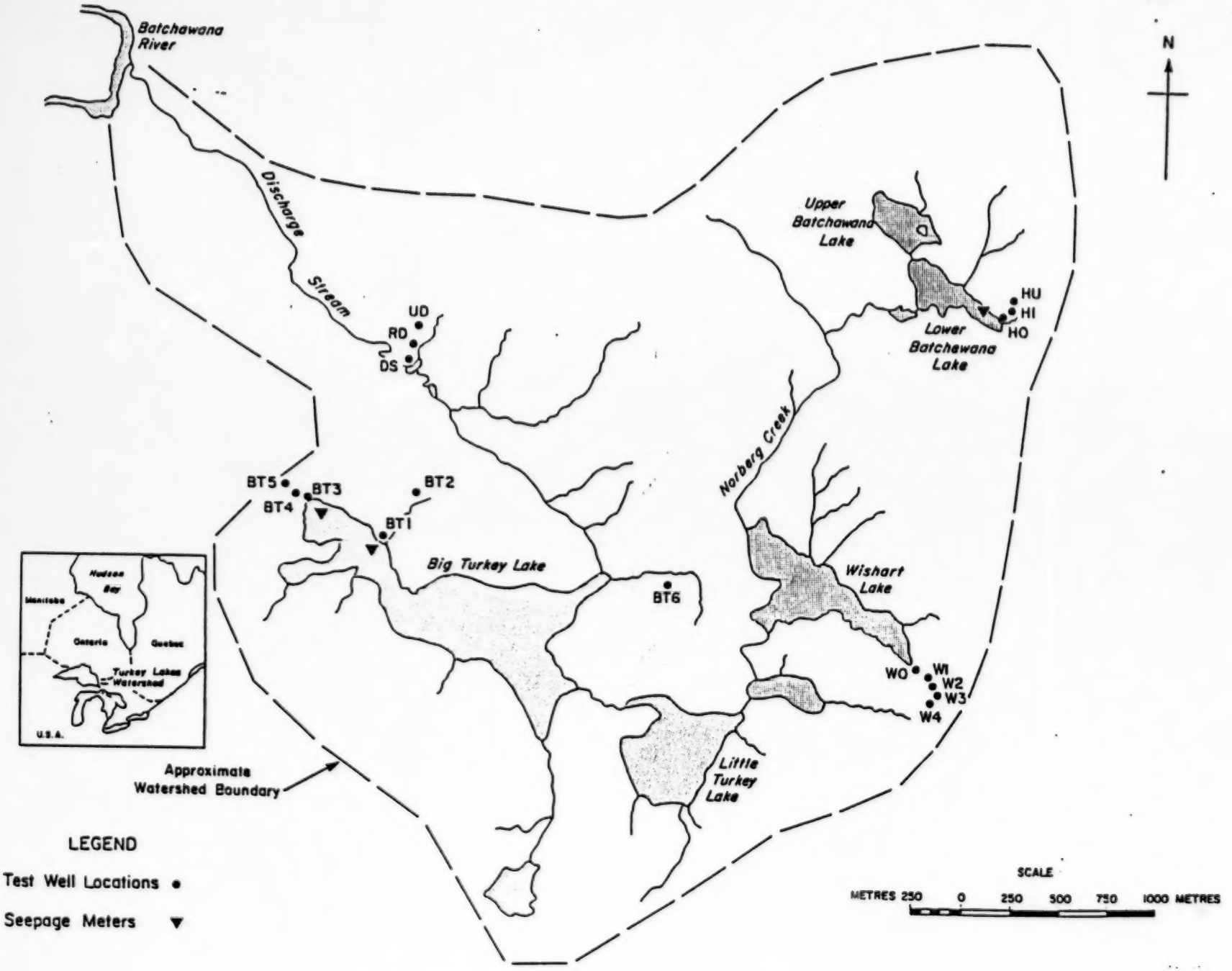
Aluminum concentration vs. time at HA, Chalk River.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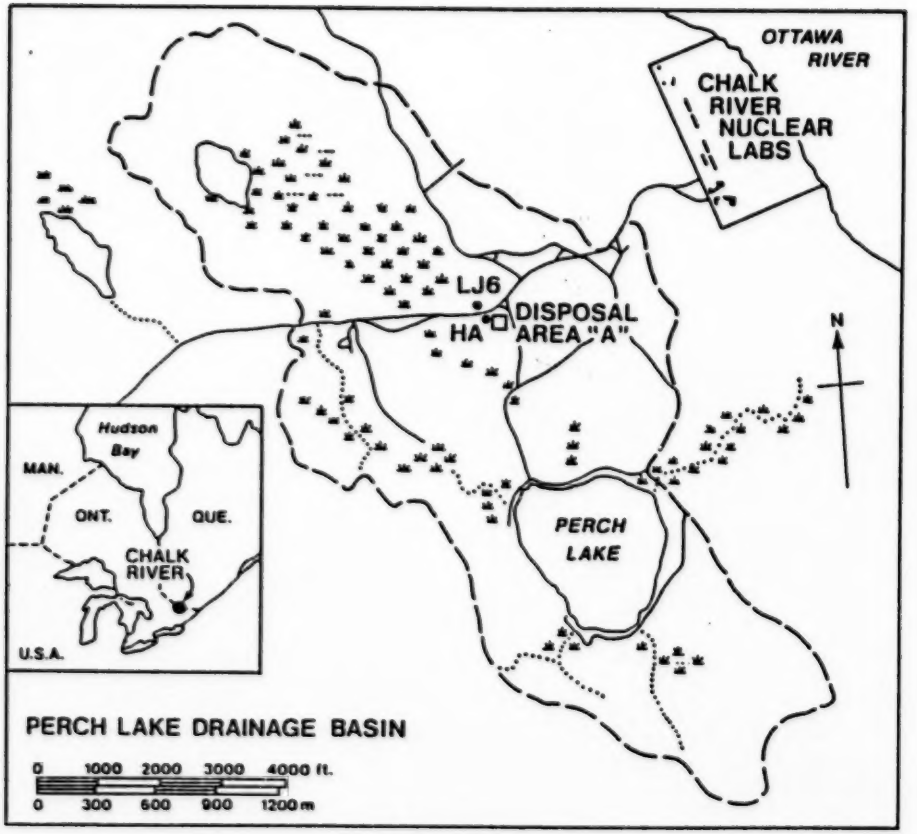
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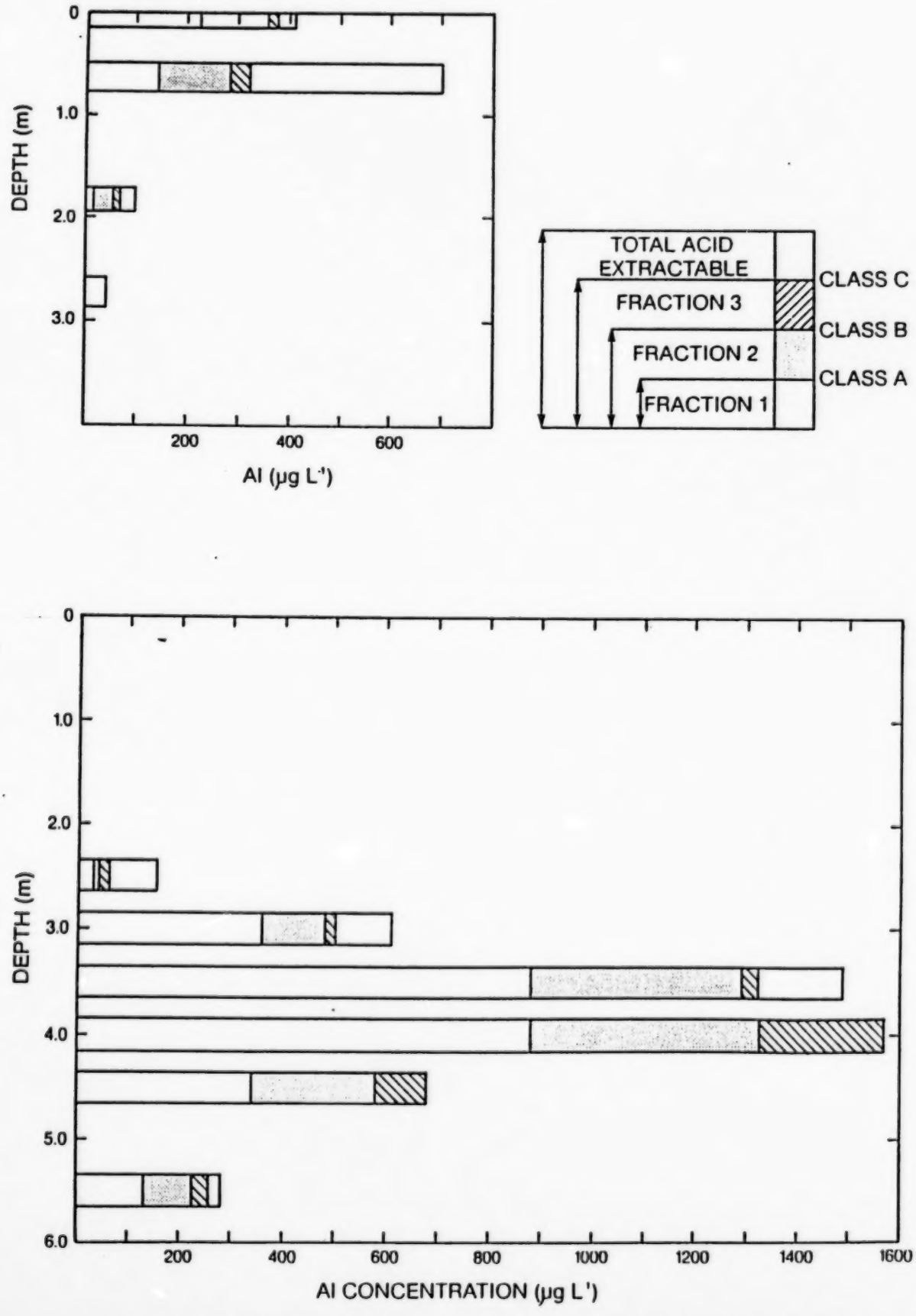
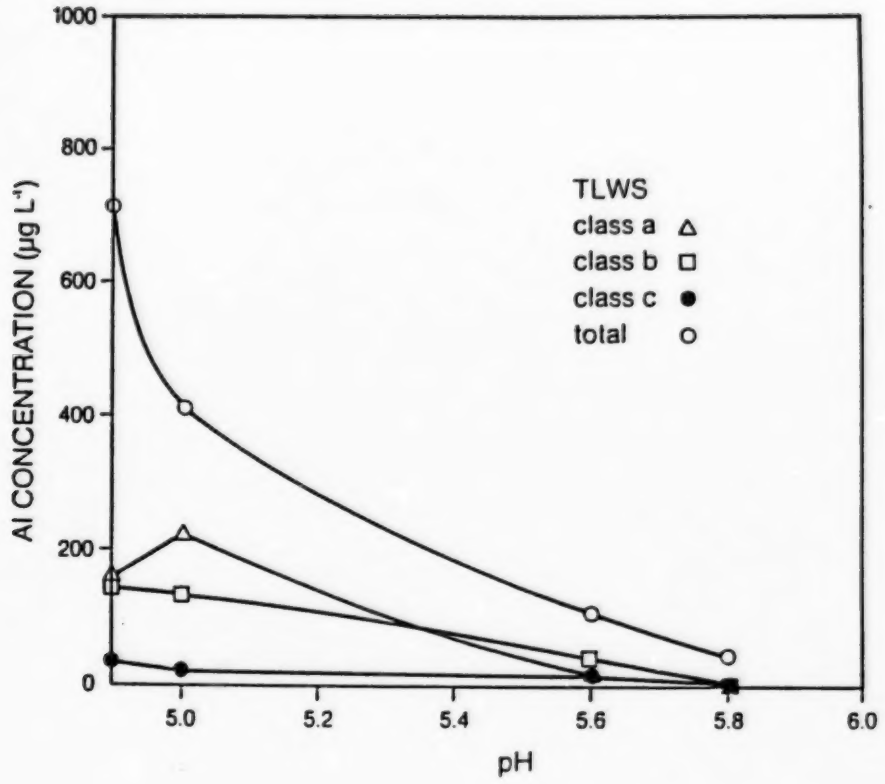



FIGURE 3: ALUMINUM CONCENTRATION VS. DEPTH AT H1, TURKEY LAKES WATERSHED, AND AT HA, CHALK RIVER.



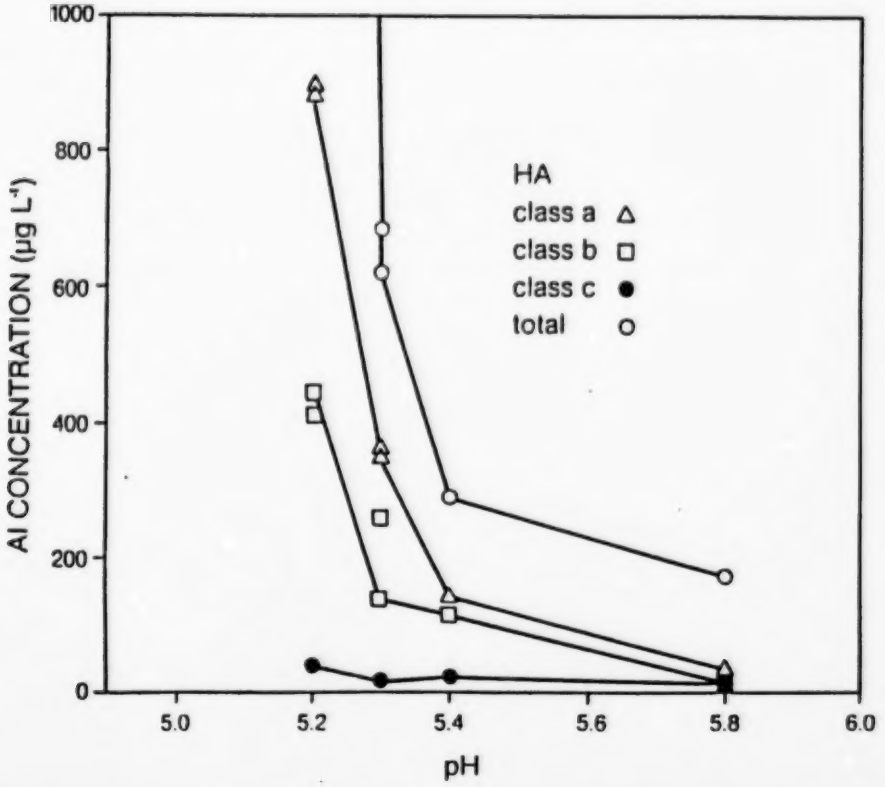




FIGURE 4: ALUMINUM CONCENTRATION VS. pH AT HU0.67, TURKEY LAKES WATERSHED, AND AT HA, CHALK RIVER

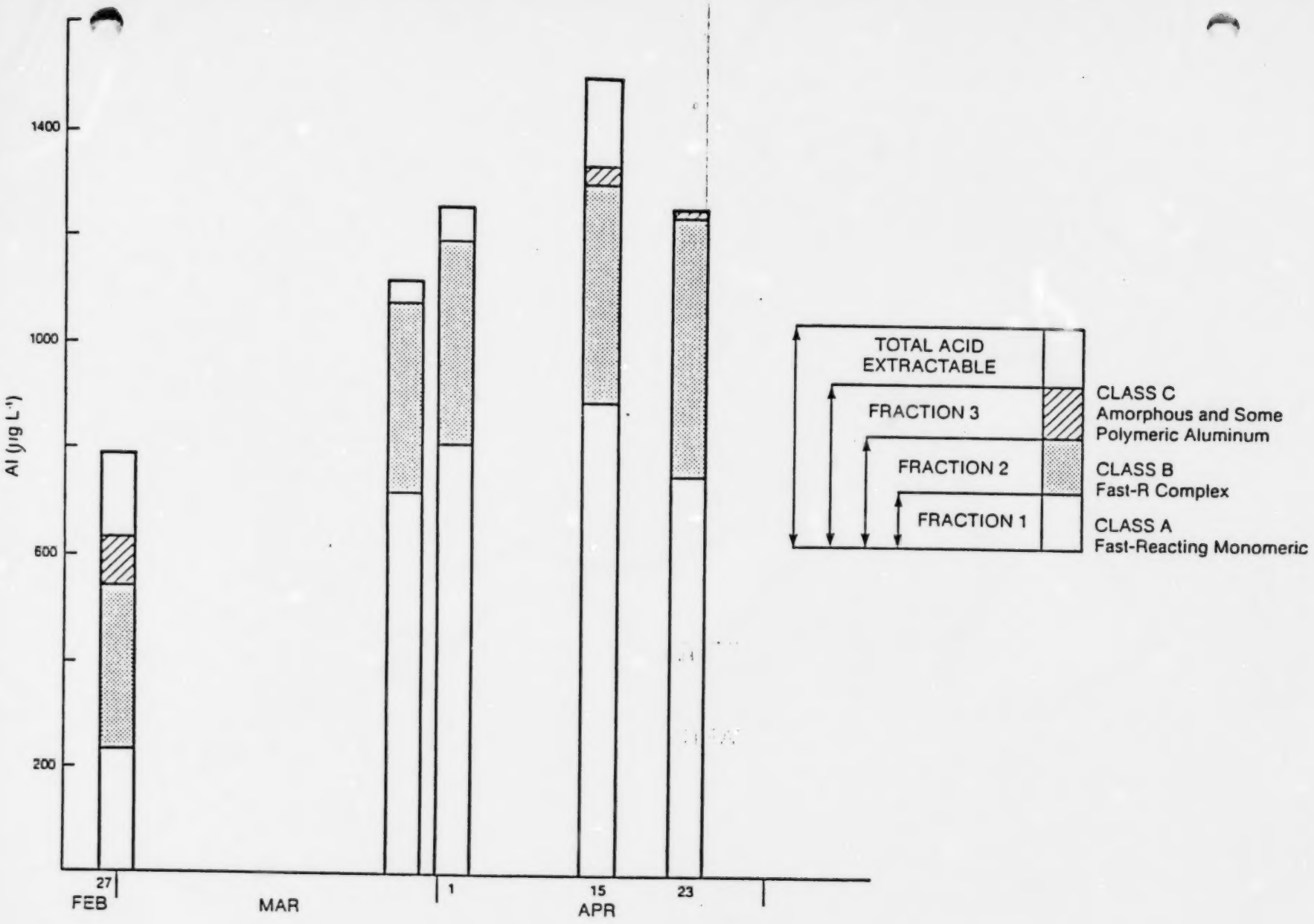



FIGURE 5: ALUMINUM CONCENTRATION VS. TIME AT HA; CHALK RIVER